Preparation of Three Coniferaldehyde Dimers and a Trimer

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Introduction

Structural elucidation of lignin by NMR relies strongly on comparison with synthetic model compounds. As described in the previous article, coniferaldehyde-containing lignins are of current interest because of the discovery of naturally CAD-deficient mutants and the current interest in antisense technology for down-regulating CAD activity in various plants. The preparation of three coniferaldehyde dimers and one trimer is described.

Method

Coniferaldehyde **1** was dissolved in acetone, and silver(I) oxide was added to the solution. The resulting suspension was stirred at room temperature for 2.5 h. The solids were filtered off, washed thoroughly with acetone and the combined solvent fractions evaporated. The resulting orange oil was separated into its components by SiO_2 thick layer chromatography. To achieve optimal separation, the plate was run several times in different solvent combinations with increasing polarity. The fractions are scraped off the plate and extracted with acetone. Three dimers **2**, **3** and **4** and one trimer **5** were isolated and have been characterized.

Conclusion and Discussion

A method has been described to prepare coniferaldehyde dimers **2**, **3** and **4** as well as the trimer **5** as a mixture, by radical reactions. It is based on radical coupling reactions effected by silver(I) oxide that have been previously utilized to prepare more conventional lignin dimers (Research Summaries 1992). NMR-spectroscopic characterization of these compounds has been completed and data are deposited in the "NMR Database of Model Compounds for Lignin and Related Cell Wall Components," available over the Internet from our sites at http://www.dfrc.wisc.edu or http://www-cwg.dfrc.wisc.edu. These compounds and their data will be used to more fully elucidate how coniferaldehyde is incorporated into plant lignins. The presence or absence of these model compounds in the polymers provides an insight into biosynthetic processes in plants and will facilitate methods for producing plants with improved digestibility.

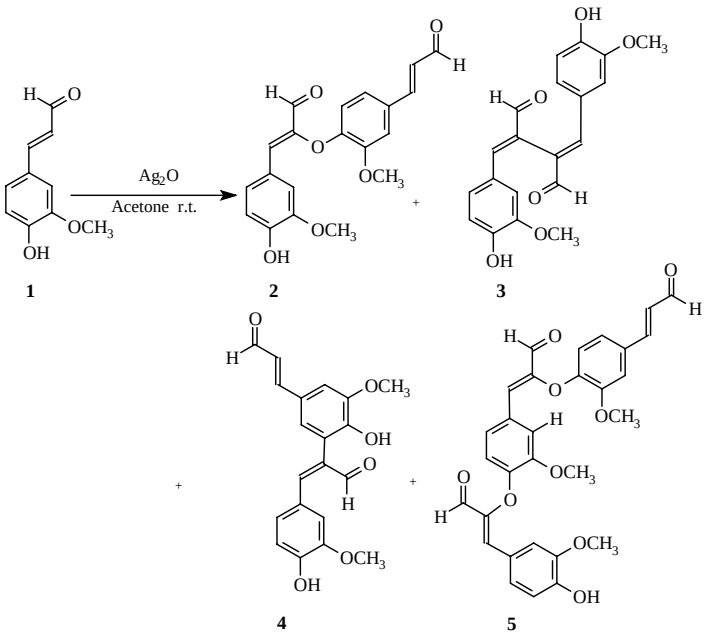

*Figure 1. Synthetic scheme used to prepare coniferaldehyde dimers **2-4** and trimer **5**.*